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METHOD FOR MAKING EMBOSSED CELLULOSE TRIACETATE FABRICS

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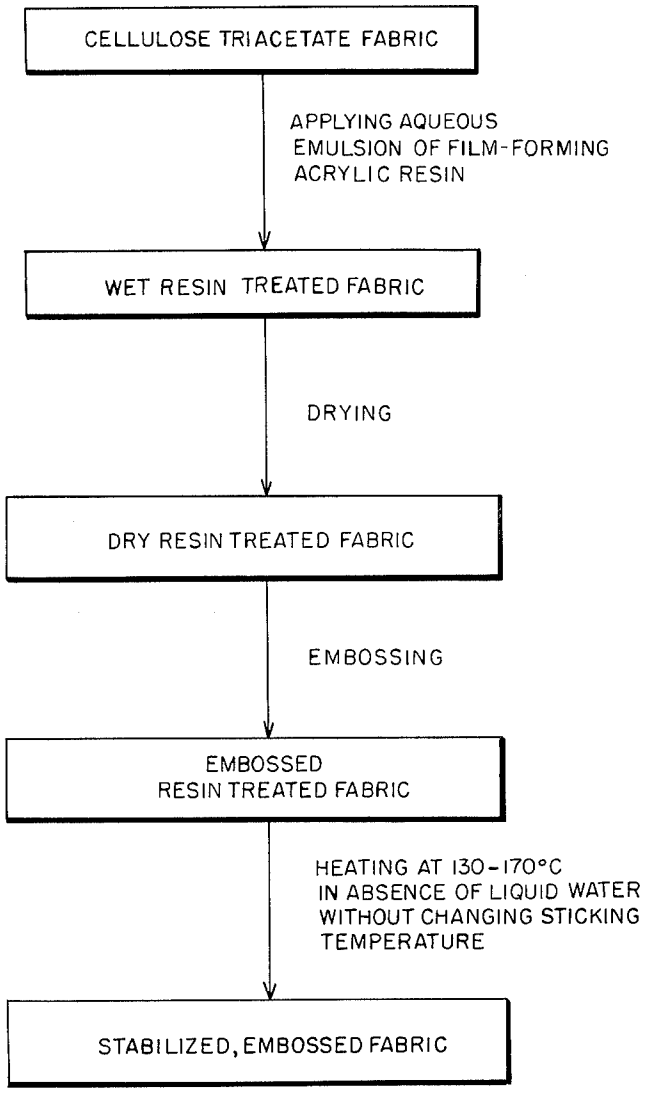

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METHOD FOR MAKING EMBOSSED CELLULOSE TRIACETATE FABRICS

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This invention relates to the production of textile fabrics and in particular to the production of embossed fabrics having a base of cellulose triacetate, that is to say cellulose acetate having an acetyl value, reckoned as acetic acid, of at least 59 percent.

Fabrics composed of fibres of cellulose triacetate may be embossed by passing the fabric through a pair of embossing rolls one of which, usually made of metal, carries the pattern it is desired to apply to the fabric and the other is made of or coated with resilient material. It has been found that the embossed configuration imparted to the cellulose triacetate fabric tends to disappear particularly after a series of washing treatments.

In order to improve the stability of the embossed configuration the embossed fabric has hitherto been subjected to a heat-setting operation that is to say, the fabric is subjected to a heat-treatment, which may be a steam treatment or a dry heat-treatment, whereby the degree of crystallinity of the cellulose triacetate is increased and the safe ironing temperature of the fabric is raised appreciably, normally to above 200° C. The heat-setting of cellulose triacetate textile materials and the nature of the improvements obtained thereby are described in the Finlayson and Krzesinski United States Patent No. 2,862,785; while such a heat-treatment improves the wash-resistance of the embossed fabric, it is found that whatever method of heat-setting is used an appreciable part of the embossed effect is lost purely as a result of the heat-treatment.

We have now found that the wash-resistance and stability of embossed effects on triacetate fabrics can be improved by subjecting the embossed fabric to a heat-treatment, preferably in the presence of one or more heat-hardenable acrylic resin or silicone film-forming compounds as described more fully below, so that the safe ironing temperatures and the sticking temperature as defined in the said Patent No. 2,862,785 are substantially unchanged by the heat-treatment.

The drawing is a flow diagram of a process according to a preferred embodiment of the invention.

Specific examples of suitable heat-treatments in the substantial absence of liquid water for use in this invention are 10 minutes at 130° C. and 1 minute at 170° C. but temperatures between 130° C. and 170° C. can be used with suitably adjusted times, for example 5 minutes at about 150° C. Temperatures above 170° C. are not recommended. It is also possible to use, as the base fabric, a fabric which has already been subjected to a heat-setting treatment before the embossing operation. In commercial operations it is often desirable to use short heating times to enable existing equipment such as stenters to be used to carry out the heating step.

In accordance with a preferred embodiment of this invention, the stabilisation of the embossed finish effected by the heat-treatment is enhanced by carrying out the heating in the presence of one or more acrylic resin or silicone film-forming compounds which have been applied to the fabric before the embossing operation. The acrylic resin is preferably a self-crosslinking resin. With such acrylic resins, and with the silicones, the heat-

treatment adopted is such as to harden or cure the resin on the fabric.

The film-forming acrylic resins which are used are polymers or copolymers of the acrylic type which may be thermoplastic polymers or copolymers of the acrylic type or may be the heat-hardenable type, that is to say they can be cross-linked by heat-treatments as specified above. The term "acrylic resin" is used to mean a polymer or copolymer containing at least 50 percent by weight of an ester, amide or nitrile of acrylic acid or of methacrylic acid, and reactive derivatives thereof. Examples of suitable acrylic esters are methyl acrylate, ethyl acrylate, methyl methacrylate, butyl acrylate and 2-ethyl hexyl acrylate, and thermoplastic polymers or copolymers of these compounds may be used in the process of this invention.

The acrylic resins which are cross-linked during the heat-treatment may be of two types, namely,

(a) A self-crosslinking acrylic polymer which contains functional groups that permit the polymer to react with itself to form a cross-linked resin, and

(b) A normally thermoplastic polymer or copolymer of the acrylic type which is mixed with a thermosetting resin such as a melamine- or urea-formaldehyde resin.

Type (a) resins consist of copolymers of acrylic compounds which include monomers which provide reactive groups which may for example be amide, amine epoxy or hydroxy groups. The reactive monomer need not itself belong to the acrylic group as defined but it must be co-polymerisable with acrylic compounds. Among such reactive monomers may be mentioned methylol acrylamide (a reactive derivative of acrylamide), N-butoxy methacrylamide (a reactive derivative of methacrylamide), 2-amino vinyl ether, ureido-vinyl ether and ureido-ethyl acrylate. A number of such self-crosslinking copolymers are at present available on the market in the form of emulsions, examples being copolymers of styrene with N-butoxy methacrylamide and ethyl or butyl acrylate. A number of the available emulsions are set out in the examples and description below.

Type (b) consists of a mixture of an acrylic type of polymer or copolymer and a thermosetting resin, the mixture being preferably made shortly before application to the fabric; on heating this mixture, the thermosetting resin effects a cross-linking of the acrylic resin.

The resin emulsion may contain any of the known types of softeners for cellulose triacetate in order to improve the handle of the fabric. Examples of such softeners are Sandozin W (Sandoz), Primenit VS (Farbwerke Hoechst), Irgavel N (Geigy Chemical Co.) and Cirrasol XL (Imperial Chemical Industries Limited).

The copolymers are applied to the fabric before the embossing operation. This may conveniently be effected by padding the fabric with an aqueous emulsion of the copolymer and drying the fabric at a temperature at which the copolymer remains substantially unchanged. The emulsion preferably contains an added catalyst such as ammonium chloride. The strength of the emulsion and the padding conditions are preferably designed so that the fabric takes up from 0.5 to 5.0 percent of its dry weight of the copolymer emulsion; a take-up of 3 percent, ±0.5 percent, has proved particularly useful.

Referring to the accompanying drawing, a preferred embodiment of the invention therefore consists in applying an aqueous emulsion of a heat-hardenable acrylic type of copolymer to a cellulose triacetate fabric, drying the fabric, embossing the fabric and then heating the embossed fabric under conditions which bring about hardening or curing of the copolymer but without substantially changing the sticking temperature of the fabric.

The heating is normally carried out at 130–170° C. and in the substantial absence of liquid water.

A further class of film-forming compounds which may be used are the silicones. Silicones, like the acrylic type of copolymers described, are heat-hardenable and may be applied to the fabric in the form of aqueous emulsions after which the fabric is dried and embossed. Particularly advantageous results have ben obtained by using a combination of an acrylic type of copolymer and a silicone in that the embossed fabric has a markedly improved wash-resistance and at the same time, mainly due, it is believed to the silicone, the fabric has an attractive soft handle. With a silicone resin the take-up is preferably in the range of 0.25 to 3.0 percent based on the weight of the fabric when dry.

The invention is illustrated by the following examples in which percentages are by weight.

Example 1

A 100 denier filament cellulose triacetate warp-knitted fabric which had previously been dyed to a dark green shade and dry heat set for 20 seconds at 205° C. was passed through the rollers of an embossing calender. The engraved steel roller, carrying a fine crepe or crystal design, was run in intimate contact with a resilient woollen paper counter bowl, a total pressure of 6 tons being applied to a 20 inch width roller (i.e. 672 lbs./linear inch). The engraved steel roller was heated to 130° C. and the speed was 3 metres/minute.

After embossing, the fabric was divided into two parts, patterns A and B. Pattern A was given a heat-treatment for 5 minutes at 150° C., whereas pattern B was not heated. Each pattern was further sub-divided into three parts, designated A–1, A–2, A–3 and B–1, B–2, B–3.

A–1 and B–1 were retained for reference.

A–2 and B–2 were washed in a laundry drum in a liquor containing 2 grams/litre of soap flakes, for 15 minutes at 40° C. using a liquor to fabric ratio of 50:1. After washing, the fabrics were rinsed and dried.

A–3 and B–3 were subjected to five washes as described above for A–2 and B–2.

Visual inspection of the six patterns showed that the heat-treatment produced only the slightest loss in definition and on subsequent washing the heat-treated fabrics A–2 and A–3 substantially retained the embossed effect, whereas the non-heat-treated fabrics B–2 and B–3 did not retain the embossed effect and were considered to be of no commercial value.

Example 2

A 75 denier filament cellulose triacetate warp-knitted fabric which had previously been dyed to a beige shade but had not been heat-set, was padded in the emulsion of a film forming acrylic resin containing reactive groups as solid under the trade name Texicryl DP/302CA (Scott Bader & Co.). The emulsion was applied at a concentration of 3 percent on the weight of dry fabric and it contained, as acid liberating catalyst, ammonium chloride at a concentration of 2 grams/litre. After padding, the fabric was dried at a temperature of 60° C. and then allowed to condition.

This fabric (reference A) and two similar fabrics designated B and C, but which had not been padded in the acrylic emulsion were each embossed under the conditions described in Example 1. After embossing, fabrics A and B were heated for 5 minutes at 150° C. whilst fabric C was not heat-treated. The heat-treatment was sufficient to cure the acrylic resin. Each fabric was then divided into four parts numbered as follows:

A–1, B–1 and C–1 were retained for reference.

A–2, B–2 and C–2 were washed once in the laundry drum as described in Example 1.

A–3, B–3 and C–3 were washed five times in the laundry drum.

A–4, B–4 and C–4 were evaluated for burst strength on the Mullen tester using a 1.2 inch diaphragm.

An untreated control (fabric D) was also evaluated for burst strength.

The degree of retention of the embossed effect after one and five washes was in the order A–2, B–2, C–2 and A–3, B–3, C–3 respectively. A–3 was superior to B–3 and C–3 was very poor and of no value. The degree of retention in the case of A–3 was excellent. There was some loss in strength on embossing and this was increased by the heating and further by the prepadding in the acrylic emulsion followed by embossing and curing.

Treatment:	Burst strength (lbs./sq. in.)
D. Untreated control	57
C–1. Embossed only	51
B–1. Embossed and heated	50
A–1. As B–1 but padded before embossing in Texicryl DP/302CA	46

The safe ironing temperature of the fabric was not affected by the heat treatment.

Example 3

A 75 denier filament warp-knitted fabric as described in Example 2 was padded at a concentration of 3 percent on the weight of dry fabric in an emulsion containing a copolymer of composition 28.1 percent N-butoxy methacrylamide, 9.6 percent ethyl acrylate and 10.8 percent styrene (ex Styrene Copolymers Limited) in the presence of 2 grams/litre of ammonium chloride. After padding, the fabric was dried at 60° C., conditioned and then embossed and heated as described in Examples 1 and 2 to cure the resin. After the heating, the fabric was divided into four parts which were tested as stated in Example 2. Using the formulation above there was very little loss of definition of the embossed effect even after five washes. The brust strength ranged from 57 lbs./sq. in. in the untreated fabric to 46 lbs./sq. in. in the treated and embossed fabric.

Example 4

A 100 denier filament cellulose triacetate warp-knitted fabric which had been dyed to dark green shade, and then dry heat set for 20 seconds at 205° C., was padded at a concentration of 3 percent on the weight of fabric of a self cross-linking, film forming, acrylic resin, Primal HA–16 (Charles Lennig & Co.) in the presence of 2 grams/litre of ammonium chloride. After padding, the fabric was dried at 60° C. and then allowed to condition. This fabric together with two pieces of the same fabric which had not been padded in Primal HA–16 were embossed under conditions specified in Example 1, with the exception that the crepe design was replaced by a roller engraved with a deep three dimensional pattern which was run in intimate contact with a woollen paper counter bowl. After embossing, the fabrics were treated as described in Example 2. The test results showed:

(a) Very good retention of deep embossed effects after five washes on the laundry drum with the fabric padded in Primal HA–16 followed by embossing and heating.

(b) Good retention to washing obtained by embossing followed by heating.

(c) Very poor retention after five washes when the embossing was not followed by a heat-treatment.

(d) In this example only small strength losses were obtained on a Mullen burst strength tester as described in Example 2, as follows:

Treatment:	Burst strength (lbs./sq. in.)
(i) Untreated control	67
(ii) Embossed only	65
(iii) Embossed and heated	64
(iv) Padded in 3 percent Primal HA–16 plus catalyst, then as (iii)	63

Example 5

Example 4 was repeated using the same type of fabric and procedure with the exception that the fabric was

padded in a 1 percent silicone emulsion MS148 (Midland Silicones) and 0.2 percent catalyst N–2230 (also Midland Silicones) based on the weight of fabric prior to embossing. Washing tests and strength tests on heated and non-heated fabrics were carried out in the manner described in previous examples. The results obtained showed:

(a) The definition of the embossed effect after embossing, but prior to heating was slightly inferior to that obtained with fabrics previously padded in self cross-linking acrylic resins as in Example 2 to 4.

(b) There was very little loss in definition on heating.

(c) After heating, the embossed effect showed good wash fastness even after five washes on the laundry drum.

(d) The fall in burst strength of the silicone treated fabric after embossing and heating was 21 percent compared with the untreated.

(e) The handle of the silicone treated fabric after embossing was soft, flowing and commercially very attractive, the fabric in this respect being superior to the acrylic resin treated fabrics.

Example 6

Example 4 was repeated using the same type of fabric and procedure with the exception that padding was carried out with an emulsion containing 3 percent of Texicryl DP/302CA, 1 percent of MS.148, and 0.2 percent of catalyst N–2230, all calculated on the weight of fabric, and 2 grams/litre of ammonium chloride. Washing and strength tests were carried out in the manner described above. The results showed:

(a) Excellent definition after embossing.

(b) Excellent definition after embossing and heating.

(c) Only slight loss in definition on heating and after five washes on the laundry drum.

(d) A smoother and softer handle than that obtained with Texicryl DP/302CA alone (Example 2).

(e) Loss in burst strength compared with the untreated control was 16.5 percent.

Example 7

Three patterns of a 100 percent cellulose triacetate fabric of construction 100 denier filament warp and 30's spun weft, and previously scoured, dyed to a green shade and dry heat set 20 seconds at 202° C., were embossed and heated as described in Example 2. Each pattern was then divided into four parts which were treated as described in Example 2, except for the last part which was evaluated for tear strength in the warp direction on an Elmendorf machine.

Examination of the treated and reference cuttings showed:

(a) Good retention of the embossed effect after five washes with the fabric padded in the acrylic emulsion, embossed and heated.

(b) Fair retention of the embossed effect after five washes with the fabric embossed and heated only.

(c) Complete loss of definition of the embossed effect even after one wash on the embossed but not heated fabric.

(d) There was some loss in tear strength on embossing, as shown by the results given below.

Treatment:	Tear strength (warp direction), grams
(i) Untreated control	576
(ii) Embossed only	448
(iii) Embossed and heated	448
(iv) Padded in 3 percent Texicryl DP/302CA plus catalyst then as (iii)	368

Example 8

A cellulose triacetate fabric having the same construction as that described in Example 7 was padded with a diluted emulsion of the product Polymer VP sold by Warwick Chemicals and understood to be a 40 percent emulsion of a copolymer of an acrylic resin and polyethylene. This copolymer is not a self cross-linking acrylic resin. The emulsion was applied at a concentration of about 3 percent on the weight of the dry fabric and it contained 2 grams/litre of ammonium chloride as curing catalyst. The padded fabric was dried at 60° C., air-conditioned and then embossed as described in Example 1. The embossed fabric was heated for 5 minutes at 150° C.

The embossed effect of the fabric was substantially unaffected after 5 washings.

Example 9

The procedure described in Example 8 was repeated but using, instead of Polymer VP, an emulsion sold by Badische Anilin und Soda Fabriken under the name Perapret HV and described as an emulsion of a self cross-linking resin with an acrylate basis. The embossed fabric obtained was substantially unaffected by 5 washings.

Example 10

The procedure described in Example 8 was repeated but using, instead of Polymer VP, a non-self cross-linking copolymer of the acrylic ester type sold by Sandoz under the name Ratifix A. The embossed fabric obtained was substantially unaffected by 5 washings.

Other film forming acrylic type resins available on the market which can be used in the process of this invention are set out below.

Resin:	Manufacturer
Ucetex T.R.	Union Chimique Belge.
Acronal 500D	Badische Anilin und soda Fabrik.
Vinacryl N4300	Vinyl Products Limited.
Turpex PA	Chemische Fabrik Pfersee.
Rhoplex B15	Rohm and Haas.

What we claim is:

1. A process for producing an embossed cellulose triacetate fabric of improved wash-resistance and stability which comprises applying to the fabric an aqueous emulsion of a film forming acrylic resin, drying the fabric, embossing the dry fabric and subsequently subjecting the embossed fabric to a heat treatment at a temperature between about 130° C. and 170° C., in the substantial absence of liquid water, without substantially changing the sticking temperature of the fabric.

2. A process as claimed in claim 1 wherein the take-up of the resin by the fabric is from 0.5 to 5.0 percent by weight based on the weight of the dry fabric.

3. A process for producing an embossed cellulose triacetate fabric of improved wash-resistance and stability which comprises applying to the fabric an aqueous emulsion of a heat-hardenable, film forming acrylic resin, drying the fabric, embossing the dry fabric and subsequently heating the embossed fabric at a temperature between about 130° C. and 170° C., in the substantial absence of liquid water to effect hardening of the acrylic resin but without substantially changing the sticking temperature of the fabric.

4. A process as claimed in claim 3 wherein the acrylic resin used is a self-crosslinking resin.

5. A process as claimed in claim 3 wherein the take-up of the resin by the fabric is from 0.5 to 5.0 percent by weight based on the weight of the dry fabric.

6. A process for producing an embossed cellulose triacetate fabric of improved wash-resistance and stability which comprises applying to the fabric an aqueous emulsion of a heat-hardenable silicone, drying the fabric, embossing the dry fabric and subsequently heating the embossed fabric at a temperature between about 130° C. and 170° C., in the substantial absence of liquid water to effect hardening of the silicone but without substantially changing the sticking temperature of the fabric.

7. A process as claimed in claim 6 wherein the take-up of the silicone resin is from 0.25 to 3.0 percent by weight based on the weight of the dry fabric.

8. A process for producing an embossed cellulose tri-

acetate fabric of improved wash-resistance and stability which comprises applying to the fabric an aqueous emulsion containing a heat-hardenable, film forming acrylic resin and a silicone, drying the fabric, embossing the dry fabric and subsequently heating the embossed fabric at a temperature between about 130° C. and 170° C., in the substantial absence of liquid water to effect hardening of the acrylic resin and the silicone but without substantially changing the sticking temperature of the fabric.

9. A process as claimed in claim **8** wherein the take-up of the acrylic resin is from 0.5 to 5.0 percent and the take-up of the silicone is from 0.25 to 3 percent, both percentages being based on the weight of the dry fabric.

10. A process as claimed in claim **8** wherein the acrylic resin used is a self-crosslinking resin.

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